

TABLE 3—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD'S AND PCDF'S—Continued

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte
	417.8253	M	$^{13}\text{C}_{12}\text{H}^{35}\text{Cl}_7\text{O}$	HpCDF (S)
	419.8220	M+2	$^{13}\text{C}_{12}\text{H}^{35}\text{Cl}_6^{37}\text{ClO}$	HpCDF (S)
	423.7766	M+2	$\text{C}_{12}\text{H}^{35}\text{Cl}_6^{37}\text{ClO}_2$	HpCDD
	425.7737	M+4	$\text{C}_{12}\text{H}^{35}\text{Cl}_6^{37}\text{Cl}_2\text{O}_2$	HpCDD
	435.8169	M+2	$^{13}\text{C}_{12}\text{H}^{35}\text{Cl}_6^{37}\text{ClO}_2$	HpCDD (S)
	437.8140	M+4	$^{13}\text{C}_{12}\text{H}^{35}\text{Cl}_5^{37}\text{Cl}_2\text{O}_2$	HpCDD (S)
	479.7165	M+4	$\text{C}_{12}\text{H}^{35}\text{Cl}_7^{37}\text{Cl}_2\text{O}$	NCPDE
	430.9729	LOCK	C_9F_{17}	PFK
	441.7428	M+2	$\text{C}_{12}^{35}\text{Cl}_7^{37}\text{ClO}$	OCDF
	443.7399	M+4	$\text{C}_{12}^{35}\text{Cl}_6^{37}\text{Cl}_2\text{O}$	OCDF
	457.7377	M+2	$\text{C}_{12}^{35}\text{Cl}_7^{37}\text{ClO}_2$	OCDD
	459.7348	M+4	$\text{C}_{12}^{35}\text{Cl}_6^{37}\text{Cl}_2\text{O}_2$	OCDD
	469.7779	M+2	$^{13}\text{C}_{12}^{35}\text{Cl}_7^{37}\text{ClO}_2$	OCDD (S)
	471.7750	M+4	$^{13}\text{C}_{12}^{35}\text{Cl}_6^{37}\text{Cl}_2\text{O}_2$	OCDD (S)
	513.6775	M+4	$\text{C}_{12}^{35}\text{Cl}_8^{37}\text{Cl}_2\text{O}_2$	DCDPE
	442.9728	QC	$\text{C}_{10}\text{F}_{17}$	PFK

(a) The following nuclidic masses were used:

H = 1.007825

C = 12.000000

 ^{13}C = 13.003355

F = 18.9984

O = 15.994915

 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

S = Labeled Standard

QC = Ion selected for monitoring instrument stability during the GC/MS analysis.

TABLE 4—ACCEPTABLE RANGES FOR ION-ABUNDANCE RATIOS OF PCDD'S AND PCDF'S

No. of chlorine atoms	Ion type	Theoretical ratio	Control limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6 ^a	M/M+2	0.51	0.43	0.59
7 ^b	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

^a Used only for ^{13}C -HxCDF.^b Used only for ^{13}C -HpCDF.

TABLE 5—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS

Compound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
Unlabeled		
Analytes:		
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDD	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25

TABLE 5—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS—Continued

Compound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
OCDF	30	30
Internal Standards:		
$^{13}\text{C}_{12}$ -2,3,7,8-TCDD	25	25
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD ..	30	30
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD ..	25	25
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD	30	30
$^{13}\text{C}_{12}$ -OCDD	30	30
$^{13}\text{C}_{12}$ -2,3,7,8-TCDF	30	30
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF ..	30	30
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDF ..	30	30
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF	30	30
Surrogate Standards:		
$^{37}\text{Cl}_4$ -2,3,7,8-TCDD	25	25
$^{13}\text{C}_{12}$ -2,3,4,7,8-PeCDF ..	25	25
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD ..	25	25
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF ..	25	25
$^{13}\text{C}_{12}$ -1,2,3,4,7,8,9-HpCDF	25	25
Alternate Standard:		
$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDF ..	25	25

METHOD 24—DETERMINATION OF VOLATILE MATTER CONTENT, WATER CONTENT, DENSITY, VOLUME SOLIDS, AND WEIGHT SOLIDS OF SURFACE COATINGS

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.
Volatile organic compounds Water.	No CAS Number assigned 7732-18-5

1.2 Applicability. This method is applicable for the determination of volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or other related surface coatings.

1.3 Precision and Bias. Intra- and inter-laboratory analytical precision statements are presented in Section 13.1. No bias has been identified.

2.0 Summary of Method

2.1 Standard methods are used to determine the volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or other related surface coatings.

3.0 Definitions

3.1 *Waterborne coating* means any coating which contains more than 5 percent water by weight in its volatile fraction.

3.2 *Multicomponent coatings* are coatings that are packaged in two or more parts, which are combined before application. Upon combination a coreactant from one part of the coating chemically reacts, at ambient conditions, with a coreactant from another part of the coating.

3.3 *Ultraviolet (UV) radiation-cured coatings* are coatings which contain unreacted monomers that are polymerized by exposure to ultraviolet light.

4.0 Interferences. [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hazardous Components. Several of the compounds that may be contained in the coatings analyzed by this method may be irritating or corrosive to tissues (e.g., heptane) or may be toxic (e.g., benzene, methyl alcohol). Nearly all are fire hazards. Appropriate precautions can be found in reference documents, such as Reference 3 of Section 16.0.

6.0 Equipment and Supplies

The equipment and supplies specified in the ASTM methods listed in Sections 6.1 through 6.6 (incorporated by reference—see § 60.17 for acceptable versions of the methods) are required:

6.1 ASTM D 1475-60, 80, or 90, Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products.

6.2 ASTM D 2369-81, 87, 90, 92, 93, or 95, Standard Test Method for Volatile Content of Coatings.

6.3 ASTM D 3792-79 or 91, Standard Test Method for Water Content of Water Reducible Paints by Direct Injection into a Gas Chromatograph.

6.4 ASTM D 4017-81, 90, or 96a, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method.

6.5 ASTM 4457-85 91, Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph.

6.6 ASTM D 5403-93, Standard Test Methods for Volatile Content of Radiation Curable Materials.

7.0 Reagents and Standards

7.1 The reagents and standards specified in the ASTM methods listed in Sections 6.1 through 6.6 are required.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Follow the sample collection, preservation, storage, and transport procedures described in Reference 1 of Section 16.0.

9.0 Quality Control

9.1 Reproducibility

NOTE: Not applicable to UV radiation-cured coatings). The variety of coatings that may be subject to analysis makes it necessary to verify the ability of the analyst and the analytical procedures to obtain reproducible results for the coatings tested. Verification is accomplished by running duplicate analyses on each sample tested (Sections 11.2 through 11.4) and comparing the results with the intra-laboratory precision statements (Section 13.1) for each parameter.

9.2 Confidence Limits for Waterborne Coatings. Because of the inherent increased imprecision in the determination of the VOC content of waterborne coatings as the weight percent of water increases, measured parameters for waterborne coatings are replaced with appropriate confidence limits (Section 12.6). These confidence limits are based on measured parameters and inter-laboratory precision statements.